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Direct growth, characterization, and optical properties of silica nanowires on cordierite monolith

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Abstract

We report the direct synthesis of amorphous silica nanowires on cordierite monolith. Our approach is based on a simple catalyst-assisted growth using Sn particles that are formed via the mechanochemical reaction of SnO_2 (s) with Al (s, l) or Si (s) during the initial phase of the growth process. The silica nanowires have a diameter of approximately 20 nm and become densely packed upon repeatedly branching and merging with each other. The silica nanowire assemblies grow up to 50 μm in length. The microscopic images reveal that the Sn catalysts govern the growth by controlling the formation of **the nuclei** on their surface. Remarkably, the Sn catalysts induce the growth of multiple silica nanowire assemblies and the formation of Sn-rich amorphous $(Si,Al)O_x$ nanotube/Al-doped orthorhombic Mg_2SiO_4 nanofiber as a minor product. The Sn-catalyzed silica nanowires show strong blue emission.
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1. Introduction

Over the past decade, nanomaterials have drawn tremendous scientific and technological interest [1]. Among them, one-dimensional (1D) nanostructures such as nanowires, nanotubes, and nanobelts are of particular importance because of their numerous potential applications [2]. Many methods have been developed to optimize the synthesis parameters and integrate 1D nanomaterials into nanodevices [3]. Catalyst-assisted growth is one of the most powerful methods for the synthesis of various kinds of 1D nanostructures consisting of carbon, semiconductors, and oxide materials [4–6]. In this approach, nanowires are mostly formed via a vapor–liquid–solid (VLS) growth process [7], in which the catalyst particles provide effective active sites that absorb gas-phase reactants and facilitate their diffusion for the consequent nanowire growth, once the supersaturation is reached. Therefore, the diameter, morphology, and structural properties of the nanowires strongly depend on the chemical and physical states, as well as the characteristics of the catalysts. For example, transition metals such as Fe and Ni are commonly utilized for the catalytic growth of carbon nanofilaments, e.g., carbon nanotubes (CNTs) and carbon nanofibers (CNFs) [8]; they have also been shown to induce the growth of group IV semiconductor nanowires [9]. Noble metals such as Au and Ag promote the growth of semiconductor and oxide nanowires (such as ZnO, GeO_x, and In_2O_3) [10–12]. Other studies have reported that Si–Fe alloy catalysts lead to the growth of Al_2O_3 nanowires [13]. Furthermore, many other catalysts have been

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developed to promote the growth of 1D nanostructures [14–16].

Silica is a largely abundant and useful material [17]. In particular, owing to the excellent optical, mechanical, and biological properties, 1D silica nanostructures are widely studied for potential applications in several fields [18–21]. As for most nanomaterials, the direct integration of 1D silica nanostructures into macroscopic substrates is one of the most important challenges for their practical use. The nanowire-based hierarchical structure has the advantage of increasing the active surface area that can react with various sources, including light for optical devices, chemical reactants for catalytic supports, and cells for bioceramics. The catalyst-assisted growth is a useful method to reach this goal. Various catalysts, such as Ag, Au, Ga, Ni, and Fe have been used for the VLS growth of 1D silica nanostructures [18,22–26]. These studies show that the morphologies, structures, and properties of as-synthesized silica nanowires strongly depend on the type of catalyst.

In this work, we directly synthesized amorphous silica nanowires on cordierite monolith via Sn-assisted VLS growth, and characterized their structure through a variety of spectroscopic and microscopic analyses. Cordierite monoliths have been widely used in practical applications as catalyst supports, absorbents, and scaffolds for cell growth [27–29]. Therefore, the search for a route that facilitates the direct growth of silica nanowires on the surface of cordierite monolith is a crucial challenge for their extended application and improved properties. As a result, silica nanowires with a diameter of approximately 20 nm were synthesized on the surface of the Sn catalysts. Various microscopic analyses were conducted to determine the configuration of the Sn-catalyzed silica nanowires and identify their growth mechanism. The results show that the Sn catalysts govern the growth by branching, merging, and packing the silica nanowires during the synthesis process. Additional observations reveal that the Sn catalysts can induce the formation of composite metal oxide nanotube/nanofiber hybrid structures. In addition, cathodoluminescence (CL) analysis was performed to examine the optical properties of the as-synthesized silica nanowires.

2. Experimental section

2.1. Synthesis of amorphous silica nanowires on cordierite monolith

50 mm × 50 mm × 10 mm (L × W × h) cordierite monolith substrates with 30 cells/cm^2 were obtained from the Honda Research Institute (Fig. S1(a)). An approximately 1-μm-thick layer was coated on the cordierite monolith substrates using a multi-metal sputtering machine (Magnetron Sputter MSP-30, Japan) (Fig. S1(b)). A 99.9% purity Al target was used; the deposition was conducted at 30 nm/min and 1.3×10^{-5} Pa for 30 min. The substrates were subsequently spray coated with SnO_2 nanoparticles to form the Sn catalysts (Fig. S1(c)). Briefly, SnO_2 nanoparticles with a size of approximately 100 nm were soaked in 10 g of ethanol to prepare a 0.03 M suspension, which was subsequently ultrasonicated for 10 min; the particles were then coated on the prepared substrates using a spray apparatus (from Iwata) with a 200-μm-diameter nozzle. The distance from the substrate to the nozzle was approximately 150 mm. The coated substrates were dried for 1 h in an oven at 100 °C and installed in the reactor. For the synthesis of the silica nanowires, the reactor was heated up to 1000 °C at 10 °C/min in Ar (99.999%; 1000 cc/min). The reactor was cooled down to room temperature once the nanowire growth was completed.

2.2. Characterization techniques

To identify the morphologies of the as-synthesized nanowires, scanning electron microscopy (SEM) analysis was performed using a field emission SEM (FE-SEM: S-4700, HITACHI) operated at an accelerating voltage of 15 keV. Osmium was coated on the samples for 10 s to obtain clearer SEM images (coater: HPC-1SW). X-ray diffraction (XRD) patterns were obtained using a Rigaku DMAX-2500 operated at 40 kV and 100 mA. The scanning range was between 20° and 55° (2θ). A Cu-Kα source emitting at approximately 1.54 Å was used for the measurement. To observe the microstructure of the amorphous silica nanowires, transmission electron microscopy (TEM) analysis was conducted using a field emission TEM (FE-TEM: FEI, Tecnai F30 Super-twin) operated at an accelerating voltage of 200 keV with a Gatan imaging filter (GIF) model 2002. For the TEM analysis, the sample was placed on TEM grids (carbon-coated Cu grids with 1.2 μm holes). Energy dispersive X-ray spectroscopy (EDX; Genesis) was used to confirm the composition and configuration of the silica and composite nanowires. To investigate the optical properties of the silica nanowires, CL spectra were obtained using a MONO CL3+ accessory (Gatan; Temperature = 300 K; spectral range = 200–820 nm; voltage = 15 keV). The TEM grid samples were used for the CL studies.

3. Results and discussion

After completing the synthesis at 1000 °C, the surface of the cordierite monolith is entirely covered with wire-like bunches having an approximate diameter of 3 μm (Fig. 1(a)). Fig. 1 (b) shows that the bunchs grow up to 50 μm in length, resembling tufts of hair. The XRD patterns of the as-prepared cordierite samples are shown in Fig. 1(c). The bare monolith sample has a typical cordierite crystal structure [30]. The XRD results obtained for the Al layer coated on the monolith are confirmed by the SEM images, which show that this layer is composed of Al particles with a diameter of 500–1000 nm (Fig. S1(b)). For the Al- and SnO_2-coated samples, the XRD patterns confirm the presence of SnO_2 nanoparticles, which are uniformly dispersed on the surface of the monoliths and cover the underlying Al particles, as shown in Fig. S1(c). After the synthesis, the diffraction peaks arising from the SnO_2 nanoparticles no longer appear in the relative XRD pattern; however, peaks corresponding to crystalline Sn can be observed. This result, which is also confirmed by the SEM analysis, implies that the SnO_2 nanoparticles are mechanochemically reduced through the reaction with Al(l) and Si(s). The presence of very weak Al_2O_3 peaks in the XRD pattern of the

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